Application Note 01557

LC/MS Analysis of Isocyanate Derivatives Using the Varian 500-MS Ion Trap Mass Spectrometer

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Introduction

Isocyanate compounds are widely used in the manufacture of pharmaceuticals, pesticides and polyurethanes. The industrial use of isocyanates is in large part based on their high reactivity toward nucleophilic agents, such as alcohols or amines. However, this reactivity also leads to high toxicity. When inhaled, isocyanates can bind to human tissues, proteins and DNA, and form toxic adducts and metabolites that may cause adverse health effects, especially to the respiratory system. Occupational exposure can occur at many workplaces while handling the native compounds, during the spraying of isocyanate-based paints, or when heating and processing polyurethane or related products.

LC/MS is the ideal tool for the analysis of isocyanate derivatives, because of its sensitivity and specificity at trace levels.

This method separates and identifies four isocyanate derivatives at low or, in some cases, sub-ppb levels. The method is completed in less than five minutes, and good chromatographic separation is achieved.

Instrumentation

- Varian 500-MS Ion Trap LC/MS/MS with ESI source
- Varian 212-LC Binary Solvent Delivery Modules
- CTC Combi PAL™ autosampler

Materials & Reagents

Five samples were supplied by OSHA and were used as received. The samples were: HDI-IC-MAMA (MW 1168.8 u), HDI-BT-MAMA (MW 1142.5 u), MDI-MAMA (MW 914.5), and HDI-MAMA (MW 611.5 u).

HPLC Conditions

Column: Pursuit™ XRs C18 3 µm, 100 x 2.0 mm ID (Varian Part No. A6001100X020)
Solvent A: Water with 0.1% formic acid
Solvent B: Acetonitrile with 0.1% formic acid
LC Program:

Time (min:sec)	%A	%B	Flow (µL/min)
00:00	90	10	400
05:00	90	10	400

Injection Volume: 10 µL

MS Parameters

Ionization Mode: ESI (positive)
API Drying Gas: 40 psi at 375 °C
API Nebulizing Gas: 50 psi
Shield: 600 V

Table 1. MS segment parameters.

Segment #	Analyte	Transition	Retention Time (min)	Needle Voltage	Capillary Voltage (V)	Ex. Amp. (V)	RF Load %
1	HDI	611.5 > 170-622	1.78	5000	95	1.2	130
2	MDI	914.5 > 246-925	2.50	4000	105	1.8	145
3	HDI-BT	1142.7 > 310-1153	3.85	4000	130	2.2	90
4	HDI-IC	1168.8 > 317-1179	4.42	5000	130	2.4	100

Results

Serial dilutions of the isocyanate derivatives were performed at room temperature in 90% acetonitrile to determine the limit of detection (LOD) for each compound. Figures 2-5 show the chromatogram plots for each compound with the respective LOD.

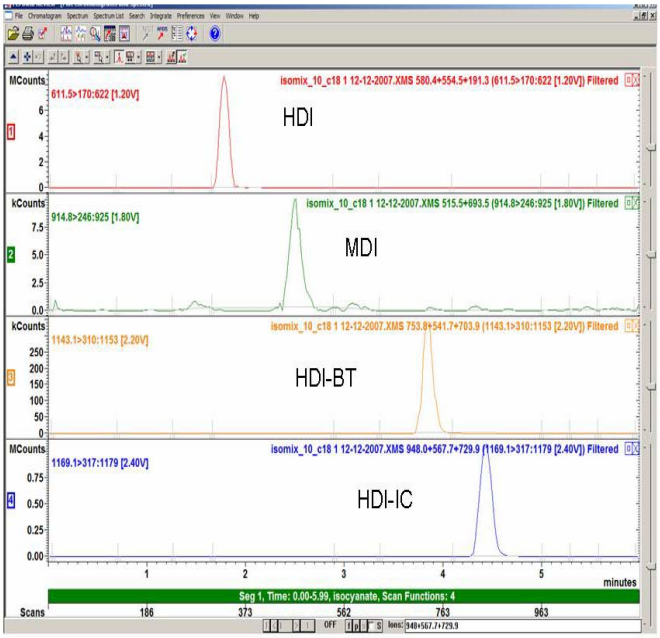

Figure 1. Extracted ion chromatogram of (1) HDI: extracted *m/z* 580.4, 554.5, 191.3; (2) MDI: extracted *m/z* 515.5, 693.5; (3) HDI-BT: extracted *m/z* 753.8, 541.7, 703.9; and (4) HDI-IC: extracted *m/z* 948, 567.7, 729.9.

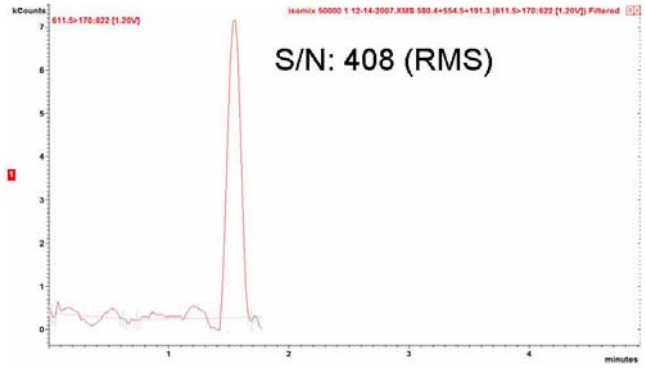

Figure 2. Chromatogram for HDI showing LOD at 75 ppt.

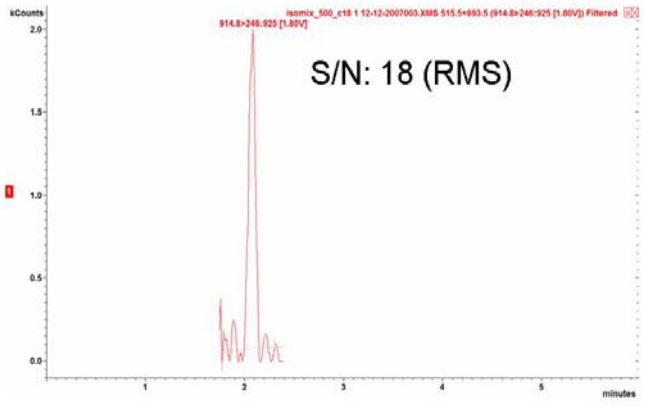

Figure 3. Chromatogram for MDI showing LOD at 5 ppb.

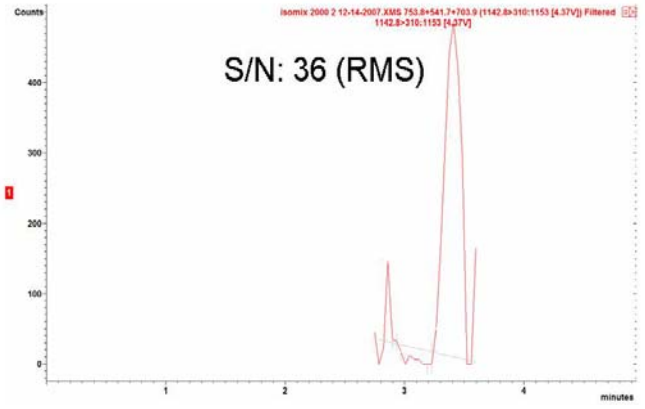

Figure 4. Chromatogram for HDI-BT showing LOD at 2 ppb.

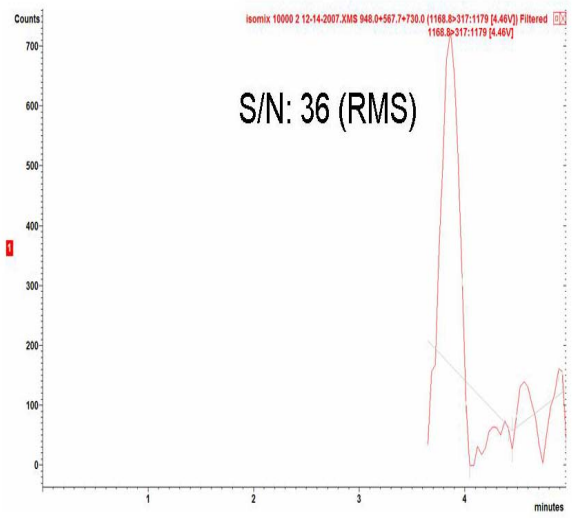

Figure 5. Chromatogram for HDI-IC showing LOD at 375 ppt.

Conclusion

The four isocyanate derivatives presented in this method were separated and identified in about five minutes.

The MS/MS capabilities of the Varian 500-MS Ion Trap Mass Spectrometer allow for isolation of desired precursor ions followed by collision-induced dissociation (CID), resulting in characteristic product ion spectra for clear, baseline resolved chromatographic peaks of the target compounds.

This method is fast, rugged and sensitive. The LOD values ranged from 75 ppt to 5 ppb for the compounds analyzed.

These data represent typical results.
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